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# QUANTITATIVE ANALYSIS OF LIQUID OXYGEN-LIQUID

## HYDROGEN COMBUSTION PRODUCTS

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QUANTITATIVE ANALYSIS OF LIQUID OXYGEN-LIQUID HYDROGEN  
COMBUSTION PRODUCTS

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SUMMARY

A method was developed for the quantitative analysis of the combustion products obtained from the firing of a liquid oxygen-liquid hydrogen rocket engine. The fixed gases hydrogen ( $H_2$ ), oxygen ( $O_2$ ), nitrogen ( $N_2$ ), and helium ( $He$ ) were determined by use of an analytical mass spectrometer. The inletting system of the mass spectrometer has been modified for high pressure sampling. Samples were obtained at the time of the rocket test run at the chamber pressure of the engine using a high pressure gas cylinder.

The concentration of water ( $H_2O$ ) was determined by a two-phase analysis. The vapor phase ( $H_2O$ )<sub>g</sub> was analyzed by a sorption hygrometer and the liquid phase ( $H_2O$ )<sub>l</sub> by weighing a tared desiccant column.

The weighing technique was cross-checked by weighing the sample cylinder "dry" as well as "wet". The water ( $H_2O$ ) was quantitatively recovered.

INTRODUCTION

Lewis Research Center engineers have been studying the performance characteristics of many chemical rocket engines. Among the studies at Lewis include a project to determine the combustion instability characteristics, namely high frequency screech of a liquid oxygen (LOX)-liquid hydrogen (LH<sub>2</sub>) engine. In support of the combustion instability program an attempt was made to analyze the combustion products of the engine to determine whether screech is a function of design or combustion. It has been found that high frequency screeching will destroy an engine within seconds of firing, and the heat transfer rates to increase from three to twelve times normal.

The subject of this report will be to discuss the development of an analytical method to quantitatively determine the combustion products of the rocket engine.

The combustion products were sampled at the engine chamber pressure using a high pressure stainless steel gas sample cylinder. The fixed gases, hydrogen ( $H_2$ ), oxygen ( $O_2$ ), nitrogen ( $N_2$ ), and helium ( $He$ ) were analyzed by use of an analytical mass spectrometer modified for high pressure sampling. The water ( $H_2O$ ) content was determined by a two-phase analysis. The vapor phase ( $H_2O$ )<sub>g</sub> was analyzed by a sorption hygrometer from 0 to 25 000 parts per million, and the liquid ( $H_2O$ )<sub>l</sub> phase by differential weight using a tared desiccant column. The weighing technique was cross-checked for accuracy by also weighing the cylinder both "wet" and "dry". Quantitative recovery of water was attained by this method.

## TEST PROCEDURE

A three-step scheme for the analysis of the combustion products is briefly outlined below:

1. Fixed gases (hydrogen, helium, nitrogen and oxygen) are analyzed by use of an analytical mass spectrometer.
2. The water vapor content determined by a sorption hygrometer
3. Liquid water measured by a weighed desiccant column

The detailed systematic analysis was accomplished as follows:

## A. Fixed Gases

1. The stainless steel gas sample cylinder containing the sample is weighed to the nearest fifteen milligrams for total weight of cylinder plus contents.
2. Sample cylinder is connected to the mass spectrometer high pressure inlet system at inlet valve,  $V_9$ , (fig. 1).
3. The inlet system was vacuum purged to the cylinder valve  $C_1$  with valves  $V_6$  and  $V_{10}$  closed.
4. Close valve  $V_7$  and vacuum valve  $V_4$ .
5. Pressurize the inlet system to valve  $V_7$  and then close valve  $C_1$ .
6. Relieve the excess pressure by slowly opening valve  $V_{10}$  and quickly closing valve  $V_{10}$ .
7. The sample gas was then introduced to the mass spectrometer using the standard operating procedure for mass spectrometer analysis.
8. The mass spectral pattern was evaluated and the concentration in mole percent is computed for each constituent.

## B. Water Vapor Analysis

1. The sample cylinder was then purge connected at valve  $C_2$  to the moisture analysis manifold (fig. 2) at valve  $V_1$  with valve  $C_2$  left open.
2. A tared "dry" desiccant column was placed between valves  $V_2$  and  $V_3$ .
3. With valves  $V_2$  and  $V_3$  closed, the water vapor content was determined by use of the sorption hygrometer.
4. A flow rate of 1500 cc/min was established by opening valves  $V_1$  and  $V_4$ , the flow maintained by the hygrometer flow control valve on the instrument.

5. When a stable moisture content was determined and recorded, the cylinder was allowed to relieve its excess pressure through the instrument, by noting the loss of flow rate on the instrument rotometer.

6. When the pressure had reached nearly atmosphere pressure, valve  $V_4$  was closed.

### C. Liquid Water Content

1. A dry helium cylinder, with the pressure regulated to approximately forty pounds per square inch gauge, was purge connected to the sample cylinder at valve  $C_1$  with  $C_1$  closed.

2. Nichrome heating tape connected to a variable powerstat was wrapped tightly around the sample cylinder.

3. The heater was turned on and controlled by the powerstat.

4. Valve  $C_1$  was opened to pressurize the cylinder with helium to approximately forty pounds per square inch.

5. Valves  $V_2$  and  $V_3$  were opened and the sample gas pressurized with dry helium to drive the liquid water from the cylinder to be trapped on the desiccant column.

6. The sample, plus helium, was allowed to flow for one hour while monitoring the moisture content of the effluent gas. This was done to insure that no water was eluted from the desiccant column.

7. After one hour, the helium was turned off and the desiccant column was isolated by closing valves  $V_2$  and  $V_3$ .

8. The cylinder was allowed to cool to ambient temperature.

9. The desiccant column and cylinder were disconnected and each reweighed and the weights recorded.

Dessicant column: A coiled 4-foot by 3/8-inch copper tube, packed with 8-mesh anhydrous calcium sulfate.

### CONCLUSIONS AND RESULTS

The use of a mass spectrometer for the analysis of the fixed gases is well established; however, the sampling of high-pressure gases (over 300 psig) without the use of a step-down pressure regulator is not routine. The high-pressure inletting system affords vacuum purging to the sampler valve without using "leaky" regulators.

The results of two typical analyses are shown in table I with the component concentrations expressed in moles of gas present.

The determination of large concentrations of water, both liquid and gas, as combustion products presented some problems.

It was essential to determine the total amount of water present. To have a sufficient quantity of sample for a moisture determination required either a very high-pressure sample or a large volume. Stainless steel high-pressure gas sample cylinders (working pressure, 5,000 psig) offered a safe means to obtain the samples at the chamber pressures required. The sorption hygrometer with a range of 0 to 25,000 parts per million of water vapor was a convenient instrument for the moisture content because it offered a short analysis time. Stable readings were obtained in one minute.

Liquid water was determined by purging the sample cylinder at 150° Centigrade with dry helium and forcing the water into a column packed with eight mesh anhydrous calcium sulfate.

The tared "dry" column was reweighed after an hour of purging and the recovery of water was quantitative. To ensure effective absorption by the column, the effluent helium was continuously monitored for moisture content. In each analysis, the water content of the effluent helium did not increase above that of the supply helium, usually 4 to 5 ppm. The reliability of this method was determined by injecting known quantities of water into the sample cylinder and performing the analysis in the described method. The results of the absorption performance of an anhydrous calcium sulfate column are reported in table II. The relative effectiveness of the weighing technique was also demonstrated by weighing the cylinder "wet and dry." The five kilogram cylinder was weighed to  $\pm 15$  milligrams by an analytical balance with a capacity of nine kilograms. The loss in weight of the cylinder was identical with the absorbed water weight of the column.

The technique for the analysis of the combustion products from a LOX-LH<sub>2</sub> system was found to be reliable and the results were adequate for the instability experiments performed.

TABLE I  
TYPICAL ANALYSES

COMPONENT GAS	RUN NO. 600	RUN NO. 605
	CONCENTRATION, MOLES	CONCENTRATION, MOLES
HYDROGEN, H <sub>2</sub>	0.303	0.266
HELIUM, He	0.0606	0.072
NITROGEN, N <sub>2</sub>	0.0021	0.00045
OXYGEN, O <sub>2</sub>	0.00019	0.000048
WATER VAPOR, H <sub>2</sub> O	0.0006	0.0006
WATER LIQUID, H <sub>2</sub> O	0.017	0.0078

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TABLE II  
PERFORMANCE OF CALCIUM SULFATE COLUMN

TRIAL NO.	WEIGHT OF WATER TAKEN, GRAM	WEIGHT OF WATER FOUND, GRAM	% WATER RECOVERY, %
1	1.0000	1.0008	100.08
2	0.5055	0.4855	96.04
3	2.0160	1.9811	98.26

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# MASS SPECTROMETER HIGH PRESSURE INLETING SYSTEM

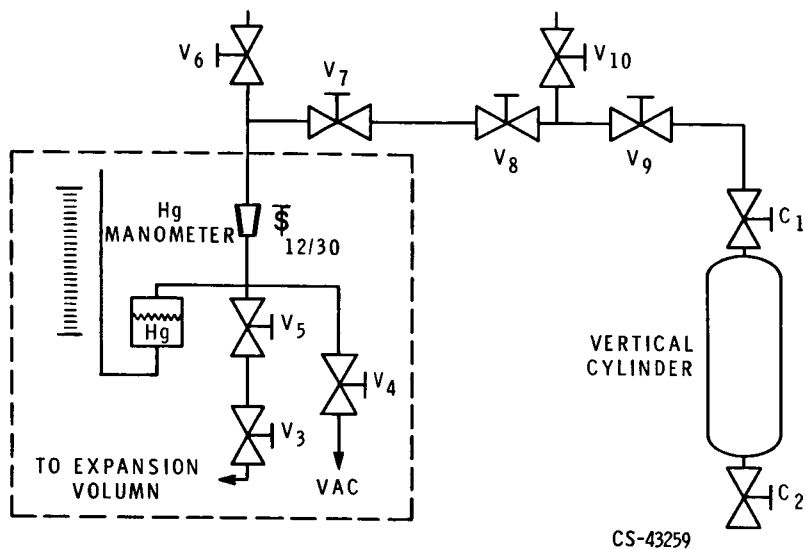


Fig. 1.

## WATER ANALYSIS SYSTEM

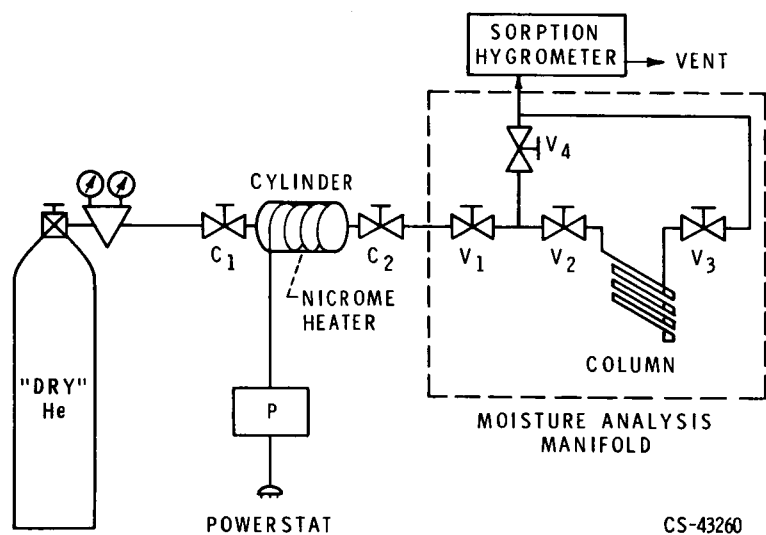


Fig. 2.